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# SURFACE BLISTERING OF METALS BY ION BOMBARDMENT

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## ABSTRACT

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Ingots of 99.999 percent pure aluminum were irradiated with mass analyzed proton and hydrogen ion beams. The irradiated materials were subsequently annealed at various temperatures for different durations and observed by photomicrography. Variation of proton energy and sample purity showed predictable results, however, the effects of sample temperature, proton flux, and oxide layer thickness have yet to be determined. The size of blisters and degree of segregation appears to be related to sample purity and degree of cold work. Additional studies involving aluminum and gold are underway.

*Author*

## FOREWORD

This report was prepared by the Avco Corporation/Tulsa Division and the Oklahoma University Research Institute as the final documentation of work performed under Contract NASw-920, issued by the Headquarters Office of the National Aeronautics and Space Administration. This program was monitored by Dr. R. R. Nash of NASA-Headquarters.

This report covers work performed from March, 1964 through April, 1965. A follow-on contract (NASw-1203) has been initiated covering the continuation of this work from April, 1965 through April, 1966.

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## I. INTRODUCTION

This is the final report prepared under Contract No. NASw-920 for a "Study of Radiation Induced Blistering of Metal Surfaces by Solar System Ions." The study has been funded through the NASA Office of Space Sciences with Dr. R. R. Nash as technical monitor. The program has been a joint venture between the Tulsa Division of the Avco Corporation and the University of Oklahoma (OU).

The program has constituted a research study to examine and define the processes which cause surface blistering of materials [irradiated by energetic ions.] Sample irradiations were conducted almost exclusively with protons. The metal which was studied during the period was aluminum, since the occurrence of surface blistering during post-irradiation annealing had been previously confirmed for this material.

This report contains a brief discussion of the background for the study, a complete review of the work performed, and a summary with tentative conclusions drawn from the results of the study. It should be pointed out that the study is being continued for another year under contract NASw-1203, also under the cognizance of Dr. Nash. Additional technical guidance will be obtained from Dr. D. L. Anderson of NASA-Ames.

## II. BACKGROUND

During the spring and summer of 1963, while the AVCO/Tulsa space environment simulation facility was in its early stage of development, work began on the "ion-metal film phenomenon." The "ion-metal film phenomenon" appeared as discolorations on metal surfaces which had been irradiated by a proton beam. Although the phenomenon itself proved to be the result of polymeric contamination, due to the seals of the primitive vacuum system then being used on the ion accelerator, several interesting research subjects evolved from it.

Since the "ion-metal film" had shown tendencies to disappear when the substrate was heated (annealed), an extensive series of proton irradiations on aluminum (6061-T6), with various subsequent heating cycles, was performed. In many cases the film disappeared, however, it tenaciously remained on others. These experiments definitely proved that the colorations were due to polymeric contamination. However, during microscopic examination (90X), a rough surface texture appeared on the irradiated portions of otherwise polished samples. Photomicrography, performed at the University of Oklahoma (OU), revealed that the surface roughening was due to a blister-like eruption of the aluminum (see Figure 1).

A few additional experiments, using an electrostatically analyzed beam, showed the reproducibility of this blister phenomenon. Figures 2 and 3 are macro and micro photos of the same piece of 6061-T6 aluminum sheet which was irradiated with  $10^{17}$  protons/cm<sup>2</sup> at 200 Kev. Subsequent annealing produced the blisters. (Note the parallel alignment of the major axis of the blisters with the roll striations in the metal.)

These experiments led to a joint research proposal by AVCO/Tulsa and OU resulting in the work reported herein. Under the terms of the contract, AVCO/Tulsa was to perform proton irradiation in their space environment simulator, while sample preparation, annealing and analysis were to be performed by personnel of the OU School of Chemical Engineering and Materials Science.

A description of the work to be ultimately performed under the study is set forth in Section III of this report.

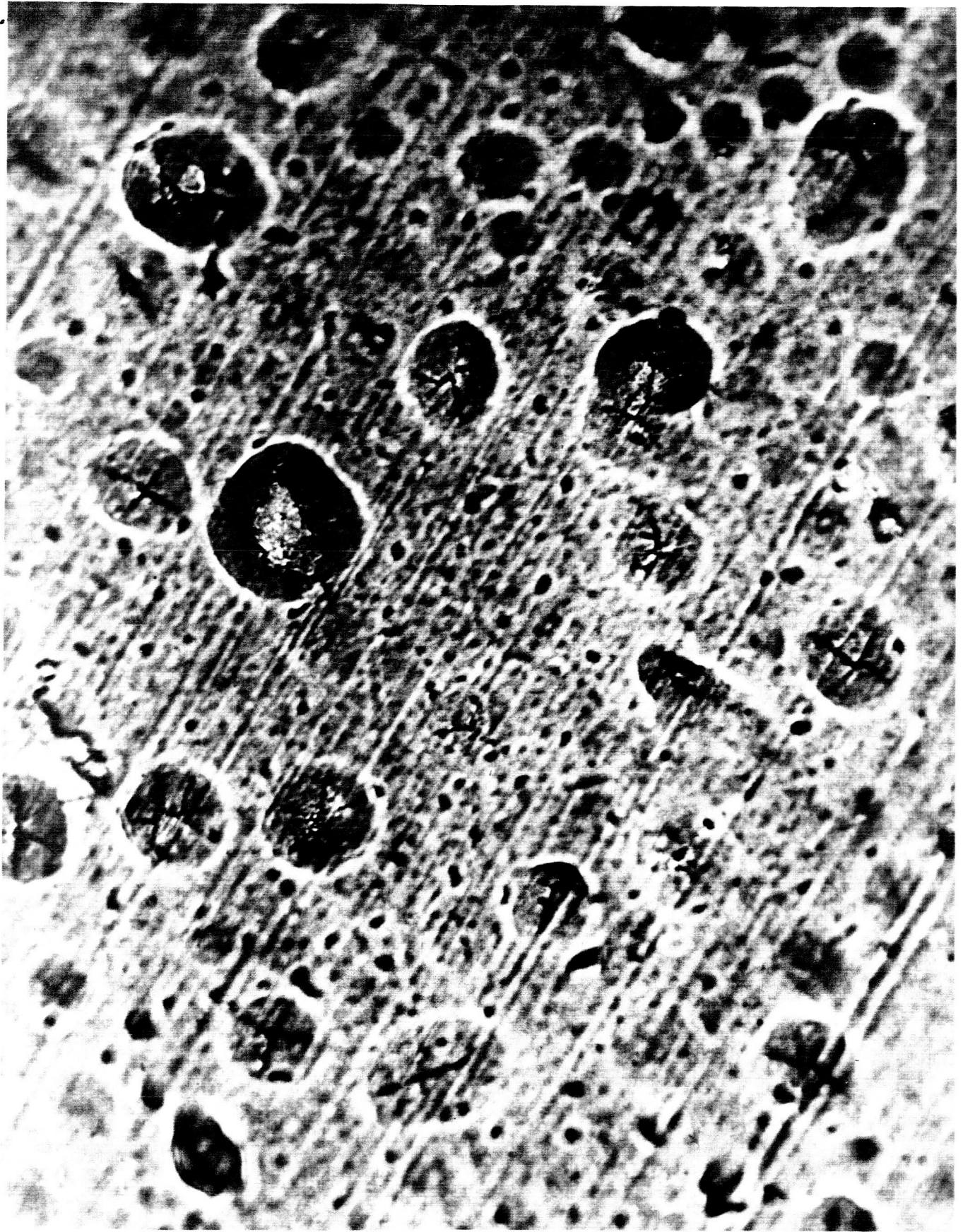


Figure 1 -- FRACTURED BLISTERS ON ALUMINUM  
(September 1963)

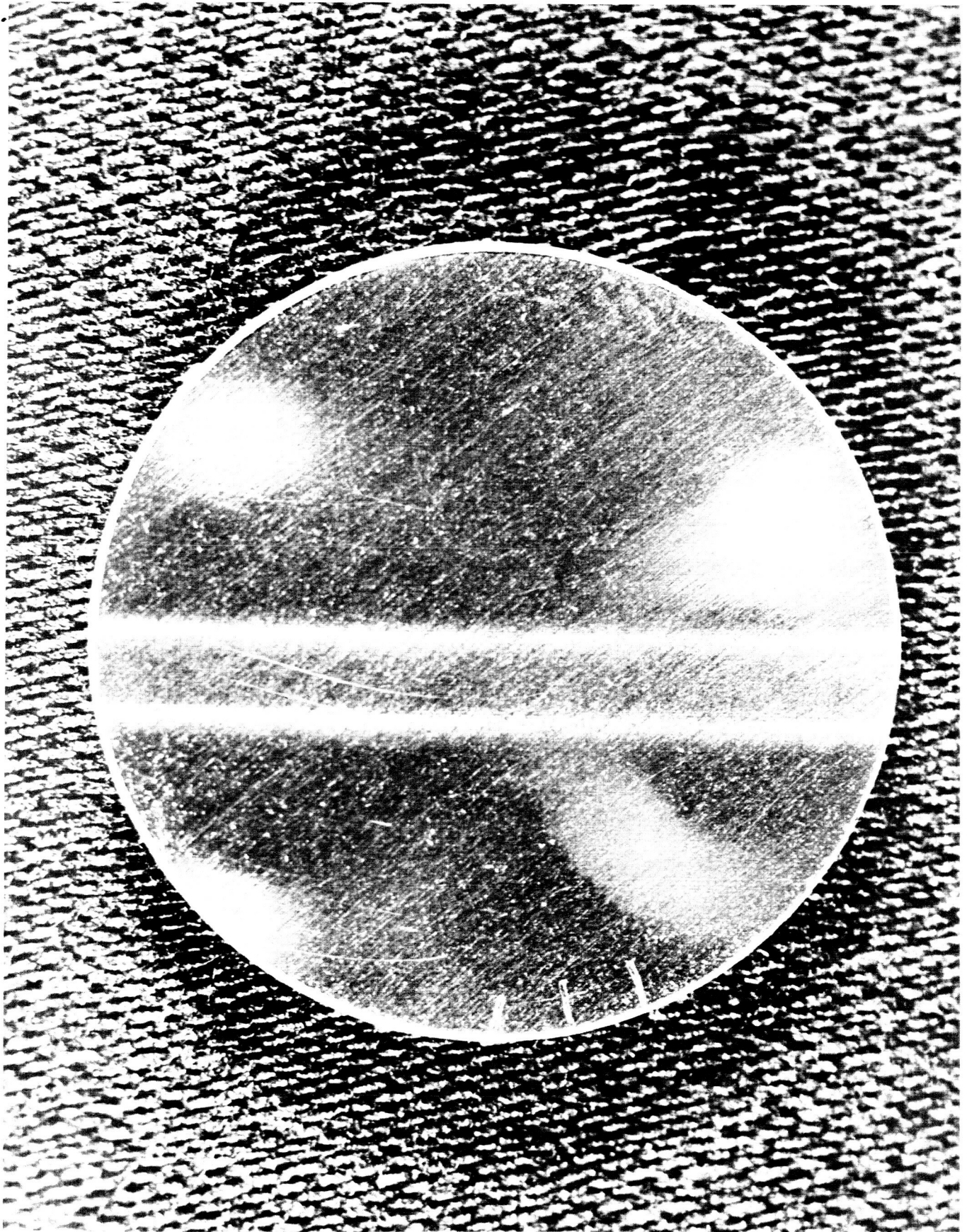


Figure 2 -- MACRO-PHOTO BLISTERS ON 6061-T6 SHEET

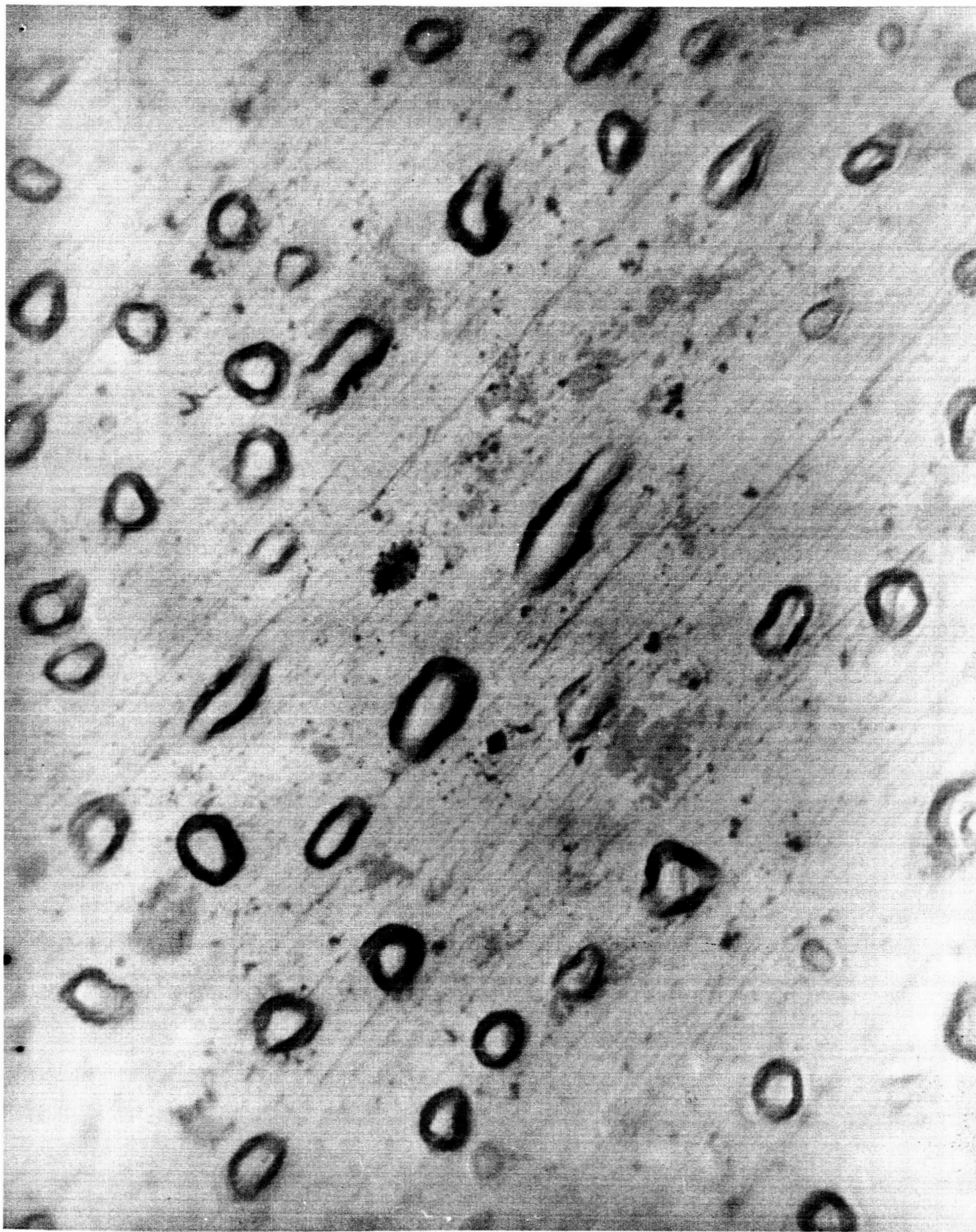


Figure 3 -- MICRO-PHOTO BLISTERS ON 6061-T6 SHEET  
(Same as Figure 2)

### III. OBJECTIVES AND PROCEDURES

It was the purpose of this program to study the processes which cause surface blistering in proton irradiated metals. Initially, the metal to be studied was aluminum since the occurrence of blistering during post-irradiation annealing had been confirmed for this material, although similar effects are expected to occur in a number of other metal and alloy systems. Surface blistering is believed to result from transport of occluded hydrogen toward the metal surface, after the hydrogen enters the metal initially as protons in an impinging ion beam.

- A. In addition to studying the reactions in the metal surface, the processes of proton trapping and the migration and agglomeration of hydrogen within the aluminum were to be investigated. Proton penetration was to be evaluated as a function of proton energy for energies of 10 Kev to about 0.5 Mev in order to correlate these studies with data on high energy experiments. The irradiating beam was to be mass analyzed in order to insure beam purity.
- B. The initial concentration and distribution of trapped hydrogen was to be studied as a function of integrated proton flux and sample temperature during irradiation.
- C. The migration and agglomeration of hydrogen was to be studied as a function of annealing conditions and the microstructure impurity of the aluminum. It was anticipated that single crystal samples and polycrystalline samples of known grain size would be used in the first phases of the program. The presence of grain boundaries is expected to affect the segregation and migration of hydrogen during annealing.
- D. Aluminum of highest purity obtainable was to be used initially for this study. Later, alloys were to be studied to evaluate the effect of impurities on hydrogen segregation and solubility in aluminum.
- E. The formation of surface blisters during annealing was to be studied by optical microscopy under an evacuated hot stage. It was hoped to correlate the formation of blisters with the kinetics of the transport process of hydrogen within the metal



and the evolution of the gas within the surface. The possibility of making concurrent observations of blister formation under the hot stage microscope and analysis of the gases evolved with a residual gas analyzer was to be evaluated.

- F. It was anticipated that the presence of surface films on the metal samples would be an important factor in the process. It was not feasible to study film-free surfaces initially, however, the thickness of the oxide film present on the aluminum could be controlled within certain limits.
- G. It was anticipated that this investigation would not only provide useful information on the conditions which lead to blister formation, but would provide significant data on the surface effects involved, the solubility of hydrogen in aluminum at low temperatures, and the transport mechanism involved in the agglomeration and evolution of hydrogen.

#### IV. EXPERIMENTAL APPARATUS

##### A. AVCO/Tulsa Simulator

The AVCO/Tulsa space environment simulator is shown in Figure 4. The simulator, as it was used in this study, consists basically of a Van de Graaff particle accelerator joined to an ultra-high vacuum chamber by appropriate plumbing.

The accelerator is capable of accelerating ions or electrons in the energy range from 10 Kev to 500 Kev. The Van de Graaff voltage generator is used from 500 Kev down to 100 Kev, below which point the voltage stability becomes poor. Energies below 100 Kev are provided by disabling the Van de Graaff voltage generator in the accelerator and connecting an auxiliary 0 - 100 Kev power supply across the acceleration tube. Positive ions are generated by an RF ion source.

The accelerator is equipped with an analyzing magnet to provide mass analysis of the ion beams and mass analyzed beams have been used consistently in the work under this contract. This contrasts with the tests performed by AVCO/Tulsa prior to the contract in which the proton beam was not mass analyzed.

The ultra-high vacuum sample chamber is pumped by a combination of sputter-ion and titanium sublimation pumping to provide an exceptionally clean vacuum environment for irradiation tests.

The accelerator and chamber are separated by a differentially pumped section to isolate the high vacuum region of the chamber from the moderately high ( $10^{-5}$  torr) region of the accelerator. The differentially pumped section is terminated at either end by slit orifices which limit gas conductance and also serve to collimate the ion beam which is then deflected to the appropriate sample positions by electrostatic means.

The sample holder which mounts in the chamber provides additional beam collimation to permit an area of 0.028 inch width across the sample diameter to be irradiated. Means are provided to control sample temperatures by flowing appropriate fluids through the sample holder.

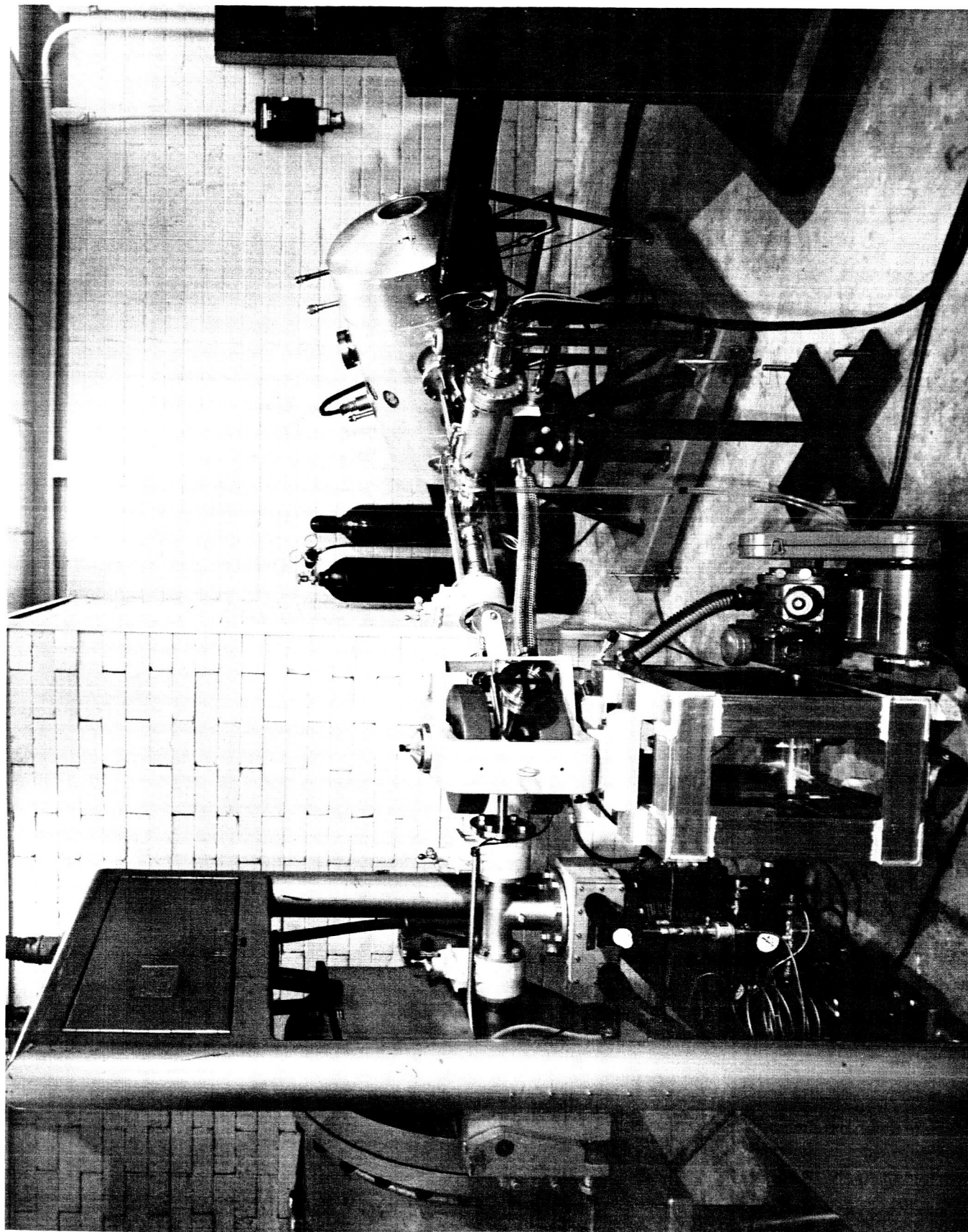


Figure 4 -- AVCO/TULSA SPACE ENVIRONMENT SIMULATION FACILITY



B. Sample Preparation and Blister Analysis Facilities

Laboratory facilities of the School of Chemical Engineering and Materials Science of the University of Oklahoma are used for the preparation of samples for proton irradiation in the AVCO/Tulsa simulator and for analysis of the irradiated samples to detect surface damage. Apparatus purchased or fabricated for this research program include a vertical traverse crystal growing furnace for melting and growing single crystal samples by the modified Bridgeman technique and a vacuum hot stage microscope assembly for simultaneous annealing and optical examination of irradiated surfaces. In addition to these specialized facilities, the metallographic preparation and examination facilities, furnace facilities, and x-ray diffraction facilities of the School are utilized in this investigation.

## V. WORK PERFORMED

### A. Sample Preparation

Materials studied in the investigation have been aluminum of 99.999% purity and an aluminum alloy, 6061-T6. The high purity aluminum, obtained in ingot form, was fabricated into samples for irradiation in the AVCO/Tulsa simulator by the following procedure: elongated pieces were cut from the ingots, swaged into long cylinders, and remelted in a cylindrical graphite crucible under an argon atmosphere. The crucible was mounted in a vertical Vycor tube and solidification of the aluminum was controlled by traversing the hot zone of a tube furnace surrounding the sample in an upward direction. Conditions for unidirectional solidification are obtained under appropriate traverse conditions and this technique was used to prepare single crystal rods of 3/8 inch diameter by about 9 inches long. Samples for irradiation, which were 3/8 inch in diameter by 1/2 inch long, were cut from the rods prepared in this manner. This sample size was dictated by the dimensions of the sample holder in the hot stage microscope and convenience in polishing the surface to be irradiated.

A longitudinal face of each sample was mechanically polished on wet abrasive laps, using 0.3 micron alumina and electropolished in a 2:1 methyl alcohol-nitric acid solution followed by rinsing in an 85 percent orthophosphoric, 3.5 percent nitric acid solution. Technique in the electropolishing process is important and critical in obtaining reproducible results. Solution temperatures, current densities and polishing times were found to be important factors in the process. It is also believed that sample purity and homogeneity are significant factors in obtaining pit-free electropolished surfaces.

Samples of aluminum alloy 6061-T6 used in the contracted study and in preliminary experiments discussed in Section II of this report were prepared by blanking out discs of appropriate size from rolled sheet. These samples were mechanically polished.

## B. Sample Irradiation and Analysis

Table I describes the samples irradiated and the irradiation-parameters used for each, along with a brief comment on the results of the post-irradiation analysis of the samples. Type of particulate radiation, total flux, particle energy, and sample temperature have been the variables examined. Although different flux rates were used, no attempts were made to correlate these. Although none of the variables were explored in great detail, a number of significant observations on conditions which give rise to surface blistering were observed.

Samples 1 through 4, which were of pure large grain polycrystalline aluminum, were irradiated under similar conditions of total particle flux, particle energy, and of sample temperature, as were corresponding samples of 6061-T6 aluminum alloy examined prior to the contract period and described in Section II of this report. The only real difference in the irradiations aside from sample material was that the pre-contract irradiations utilized unanalyzed particle beams and all samples in Table I were irradiated with mass analyzed beams. A study of the unanalyzed beam revealed that it consisted of approximately 50% protons, 48.5%  $H_2^+$  and  $H_3^+$  ions (roughly equally divided), and about 1.5% heavier ions.

Following irradiation, samples 1 through 4 were air annealed according to the following conditions:

200° C to 500° C temperature range.  
50° C temperature intervals.  
30 minutes at each temperature.

The samples were examined under the microscope after each 30 minute anneal. No blistering was observed on any of the samples. On samples 2 and 3 a darkening of the surface occurred in the irradiated area after annealing at 300° C. This darkening persisted up to a temperature of 400° C but disappeared during the 400° C anneal.

Two samples of 6061-T6 alloy (denoted here as A and B), which had been irradiated prior to the contract initiation but which had not been previously annealed, were subjected to annealing. Irradiation conditions for these samples were similar to those

TABLE I

Sample No.	Particle Type	Particle Energy (Kev)	Total Flux (p/cm <sup>2</sup> x 10 <sup>17</sup> )	Flux Rate (p/cm <sup>2</sup> /sec. x 10 <sup>12</sup> )	Sample Temp. (°C)	Comments
1	Protons	200	1	3.6	100	No Blistering
2	Protons	200	1	3.6	15	No Blistering
3	Protons	200	1	3.6	-196	No Blistering
4	Protons	200	0.1	3.6	15	No Blistering
5	Protons	200	1	2.9 (3 hr), 5.4 (3.6 hr)	15	No Blistering
6	Hydrogen H <sub>2</sub> <sup>+</sup>	200	0.5	1.8	15	No Blistering
7	Hydrogen H <sub>3</sub> <sup>+</sup>	200	0.35	1.44	15	No Blistering
8	Protons	100	1	4.3	15	Blistered
9*#	Protons	200	1	3.6	15	No Blistering
10	Protons	50	1	3.6 (1 hr), 5.4 (4.5 hr)	15	Oxide Removal
11	Protons	50	1	5.4	15	Oxide Removal
12	Protons	50	1	5.4	15	Oxide Removal
13	Protons	100	1	10.8	15	Blistered
14	Protons	100	1	10.8	15	No Blistering
15	Protons	100	1	10.8	15	No Blistering
16	Protons	100	1	10.8	15	Blistered
17	Protons	100	3.5	12.6	15	Blistered
18	Protons	100	1	10.8	15	Blistered
19	Protons	100	1	10.8	15	Blistered
20	Protons	100	1	10.8	15	Blistered
21	Protons	100	1	10.8	15	Blistered
22	Protons	100	1	10.8	15	Blistered
23#	Protons	100	1	10.8	15	Blistered
24	Protons	100	1	10.8	15	Blistered
25	Protons	100	1	10.8	15	Blistered

## SAMPLE IRRADIATIONS

\*6061-T6 aluminum alloy. All others 99.999% pure aluminum.

#Mechanically polished. All others electropolished.

/Sample surface too rough to identify blistering.

for samples 2 and 1, respectively, except for the unanalyzed beam. At 250° C, sample A darkened in the irradiated area and blisters were observed at a magnification of 300X. This sample was subsequently aged for a total of six hours at 300° C and for 10 minute intervals at 400, 450 and 500° C. There was no change in blister concentration or size. Annealing had no effect on sample C. The results were in agreement with those observed in the pre-contract tests.

The failure of the 99.999% pure aluminum samples to exhibit surface blistering in contrast to the alloy samples which were irradiated under virtually the same conditions prompted the choice of irradiation parameters and the choice of one alloy sample in samples 5 through 9. Samples 5 through 8 were very large grain polycrystalline samples of the pure aluminum while sample 9 was of 6061-T6 alloy. The possibility existed that the  $H_2^+$  and  $H_3^+$  ions were primarily responsible for blistering occurring on the alloy samples following irradiation with 200 Kev particles because of their reduced depth of penetration. On the other hand, material impurities and/or surface conditions resulting from the different mode of surface preparation (mechanical vs. electropolish) could have played a significant role.

Samples 5 through 9 were air annealed following irradiation. No blistering or change of luster of the irradiated area was found in samples 5, 6 and 7. Annealing of sample 8 for 30 minutes at 250° C produced a change in luster of the irradiated area visible to the naked eye, but no change could be seen under the microscope. Further annealing at 300° C for 10 minutes produced a very fine distribution of blisters visible under the microscope. No blistering or change in surface luster of the irradiated area was observed for sample 9 after 30 minute anneals at 250 and 300° C. Ten minutes at 350° C produced a faint line visible only with the naked eye. This line became very distinct after a 10 minute anneal at 400° C and disappeared completely within 20 minutes at this temperature.

The surface blistering which developed in the course of annealing sample 8 is shown in Figure 5 (magnification 310X). The most impressive thing about the observed blistering is the



Figure 5 -- BLISTERED SURFACE (Note Hexagonal Arrangement)

overall hexagonal pattern of the blisters. The blisters formed in a manner similar to that experienced with the aluminum alloy with regard to temperature and time at temperature, but the blisters were considerably smaller in the pure aluminum.

The hexagonal arrangement of the blisters is believed to be caused by the substructure of the metal surface. The irradiated sample was cut from a length of rod grown from the melt. Although the sample was not a single crystal, it was of such large grain size that the conditions of unidirectional solidification would still apply. The phenomenon of a cellular substructure running parallel to the growth direction has often been observed under conditions of unidirectional solidification of crystals from the melt. The substructure is believed to be caused by segregation of impurities to selective regions of the material during solidification. It is not known how this non-uniform composition affects the formation of blisters, but it appears that a greater concentration of blisters occurs in the cell boundaries which are expected to be impurity-rich regions. It is reasoned that the agglomeration of protons to form hydrogen bubbles in the lattice or blisters in the surface may be easier in the presence of impurities because of a lowered surface energy, or a greater concentration of defects in the region of impurities may provide paths for rapid diffusion of hydrogen with subsequent higher concentration of blisters in these regions.

Single crystal samples 10 through 12 were irradiated with 50 Kev protons, other conditions being the same as for sample 8. Visual examination of these samples revealed an apparent darkening of the irradiated area. Examination of the areas under microscope revealed a broken and non-uniform oxide layer. The degree of irregularity in the oxide layer varied among the samples from complete elimination to almost complete retention. Figure 6 shows a typical area where particle oxide removal has occurred. The exact mechanism of oxide removal is not known as yet. Some sputtering can occur at this energy but the erosion experienced here primarily appears to take the form of breaking up of the oxide layer into microscopic pieces with subsequent removal (see Figure 6). This mode of surface layer removal has been noted previously by AVCO/Tulsa during proton irradiation of barrier-anodized aluminum samples even on a macroscopic scale.

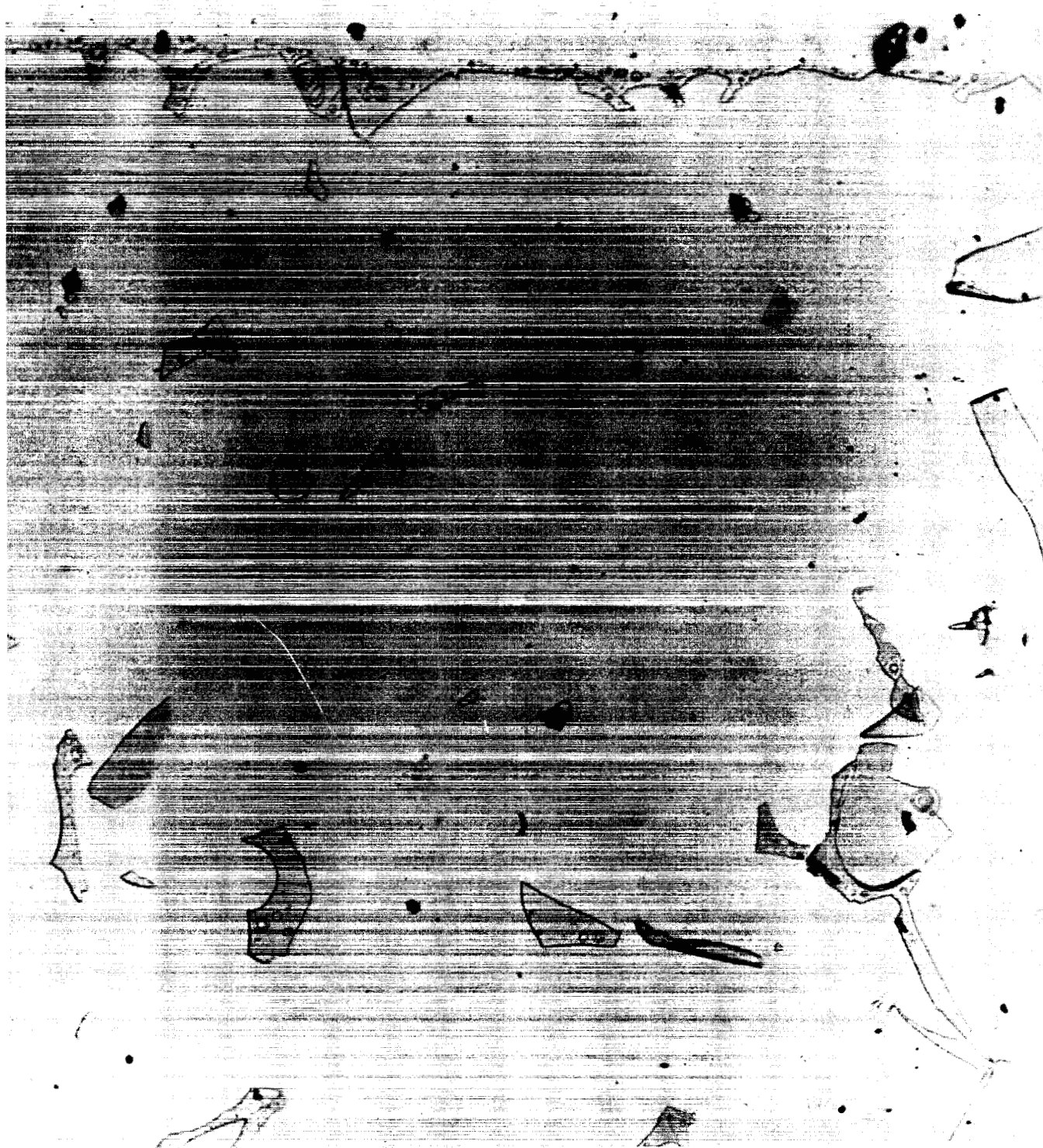


Figure 6 -- NATURAL OXIDE LAYER REMOVED BY PROTON BOMBARDMENT  
(Pure Aluminum)

Samples 13 through 25 were all very large grain polycrystalline samples of the high purity aluminum. They were all irradiated with 100 Kev protons under identical conditions except for sample 17 which was irradiated at a higher rate and with a higher total flux.

Samples 13 through 17 were prepared in the usual manner by electropolishing. Sample 13 exhibited no blistering or apparent change of surface luster during air annealing. Samples 14 and 16 exhibited a distribution of fine blistering with no tendency for segregation. Air annealing produced a faint darkening of the irradiated area, visible only to the naked eye on sample 15, but this disappeared under further annealing. No blistering occurred. Sample 17 exhibited the same random distribution of fine blisters as did samples 14 and 16, except that large blisters formed in a portion of the irradiated area where severe scratching of the surface had occurred.

Samples 18 through 22 were all air annealed for three hours at 550° C prior to electropolishing and prior to irradiation. Air annealing of samples 18, 19 and 21 produced a random distribution of fine blisters in the irradiated area. Sample 20 blistered throughout the irradiated area but with a heavier concentration along lines which appear to be due to impurity segregation but in a pattern different from that of sample 8 (Figure 5). On sample 22 blisters formed in a hexagonal formation similar to sample 8 although the pattern was not as definite.

Figure 7 shows an electron microscope replica of a portion of the blistered area of sample 21 with 31,000X magnification. The figure shows clearly the random distribution in both size and position of the blisters which have an estimated height of about two microns. Apparent small cracks in the oxide are also visible. The source of the flat areas on the blisters is unknown but they may have occurred when the replica was made.

Sample 23 was prepared with a mechanical finish followed by a three hour 550° C air anneal prior to irradiation. The surface of this sample was so rough as to preclude identification of surface blistering.

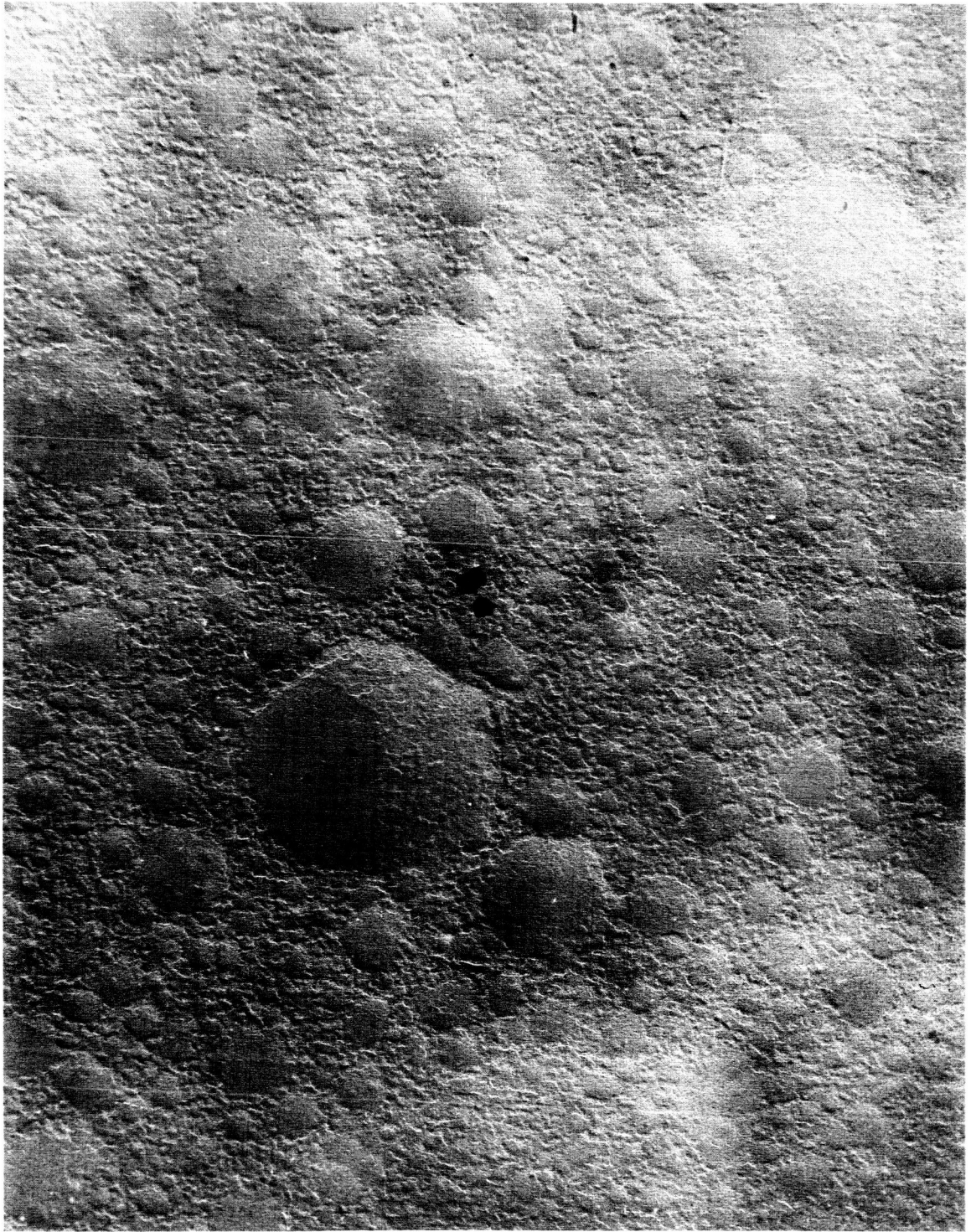


Figure 7 -- ELECTRON MICROSCOPE REPLICA -  
Sample No. 21



Sample 24 was cut from the tip of a grown crystal while sample 25 was cut from the opposite end of the crystal. These samples were electropolished but not annealed prior to irradiation.

Following post-irradiation annealing, sample 24 exhibited blisters in a roughly hexagonal pattern in portions of the irradiated area. Sample 25 also showed a segregated blister pattern, but in this case the pattern was similar to that of sample 20.

## VI. SUMMARY AND CONCLUSIONS

Twenty-four samples of aluminum grown from 99.999 percent pure aluminum ingot, and one sample of 6061-T6 aluminum alloy (see Table I) were irradiated with mass-analyzed hydrogen ion beams in the AVCO/Tulsa space environment simulator. The samples were then analyzed at the School of Chemical Engineering and Materials Science of the University of Oklahoma in a study to determine the processes which cause surface blistering to occur on proton irradiated metals during high temperature annealing. The high purity aluminum samples studied were of large grain polycrystalline structure, some with large enough grains to be evaluated as single crystal surfaces.

The effects of sample temperature during irradiation, irradiation by ions other than protons, and variation of irradiation rates and integrated particle fluxes have not been explored sufficiently to draw any conclusions. Only the effects of proton energy, and to a certain extent of sample purity and surface conditions, have provided information leading to any conclusions, and these conclusions must be considered as tentative.

At 200 Kev proton energy there has been no apparent alteration of the surfaces of any of the aluminum samples either before or after annealing. This is by contrast to the 6061-T6 alloy samples (see Figure 1) irradiated with unanalyzed 200 Kev protons prior to the work under the contract.

When samples were irradiated with 50 Kev protons, surface cleaning occurred in the form of oxide layer removal primarily as microscopic pieces. The exact removal mechanism is not known with certainty as yet. No blisters were visible on the surface either before or after annealing.

Following irradiation with 100 Kev protons, blistering occurred on most samples when they were annealed in air in the 250 to 350° C temperature range.

The apparently strong dependence of the blistering phenomenon on proton energy suggests an equally strong dependence upon the thickness of the surface oxide layer on the samples. The degree of dependence remains to be determined. It may be a total

dependence, i. e. , it may always be the oxide layer that is blistering rather than the aluminum substrate. On the other hand, the oxide layer may only limit the depth of penetration of the protons and thus the degree of blistering.

A number of effects have been observed in the formation of blisters to indicate that sample purity and preparation are important factors in determining sensitivity of a sample to blistering. The size of blisters and their degree of segregation appear to be related to sample purity and/or state of segregation and cold work in the sample. Blisters found on 6061-T6 alloy samples are larger than those found on the high purity aluminum. The blisters on the alloy are elongated in the direction of rolling (see Figure 3). The blisters found on single crystal surfaces of high purity aluminum prepared by unidirectional solidification show a degree of segregation to what are believed to be subgrain boundaries or a cellular substructure (see Figure 5). This tendency for segregation at first appeared to be eliminated when the grown crystals were annealed at 550° C prior to being irradiated with protons. However, the results of further tests on pre-annealed samples were inconsistent with this conclusion.

Also, it is observed that blisters found on the surface of a high purity aluminum sample containing a region cold worked by gouging are larger in the cold worked region than elsewhere and are oriented with the directions of the deformation (see Figures 8-A and 8-B). Figure 8-A was taken after a 20 minute anneal at 250° C and Figure 8-B after a 10 minute anneal at 350° C. These figures represent substantially the same area on the sample. Of particular interest is the alignment of the blisters along the gouge marks and the greater blister density following the higher temperature anneal. This was the only sample for which such an increase in blister density with increased annealing temperature was noted.

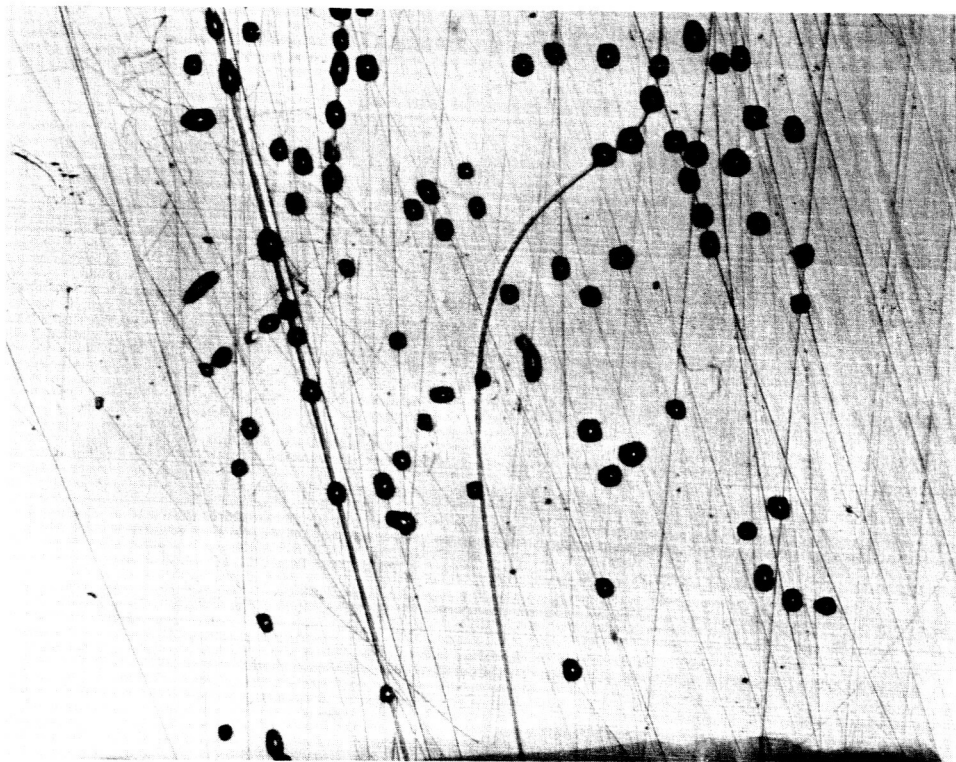


Figure 8-A -- BLISTER FORMATION  
(250° C for 20 Minutes)



Figure 8-B -- BLISTER FORMATION  
(350° C for 10 Minutes)

VII. LITERATURE SEARCH

A literature search was conducted in an effort to find information relevant to surface blister formation or apparently related phenomena. Numerous articles have been found and a bibliography of some pertinent articles is listed below.

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## VIII. FUTURE WORK

### A. Oxide Layer Studies

Since it is suspected that the presence of surface films on the aluminum samples is an important factor in the blister formation process, initial work in the follow-on study will be directed toward defining the role(s) of these films. This work will involve studies with samples from which the oxide layer has been removed in the vacuum chamber prior to the proton bombardment. Low energy ions, e. g., 50 Kev protons, will be examined first as a mechanism for removing the oxide, since surface cleaning has occurred with such protons with no apparent residual blistering. Other studies will be conducted with films of known oxide thickness.

In conjunction with these tests, studies will be pursued with pure gold samples to see if blistering occurs for this metal which is not reactive with oxygen.

### B. Material Purity and Preparation

The factors in the composition and preparation of the aluminum samples which influence blister formation will be investigated. Electron microscope replicas of blistered surfaces will be obtained to study the structural features of the fine blisters obtained in high purity material. The interference microscope will be used to determine the contour and height of blisters.

Aluminum alloy samples will be prepared by melting techniques similar to those used with high purity aluminum and the effects of composition and segregation will be evaluated. It is anticipated that electron microprobe analyses will be obtained across regions of samples with blister segregation to determine if impurity segregation is a causative factor. A furnace for melting and growing of single crystal samples under ultra-clean vacuum conditions is being designed and will be employed for material preparation. Blister formation under various annealing conditions will be compared for single crystals and polycrystalline samples.



The effect of cold work on blister formation will be studied by irradiating samples with various degrees of cold reduction and various orientations of the deformed grain structure.

C. Radiation Type and Energy

Initially, the studies will be continued with protons at 100 Kev energy since blister formation has reproducibly occurred with these particles. When the effects of oxide layers, material purity and preparation are understood, the energy and type of irradiating particles will be varied.  $H_2^+$ ,  $H_3^+$  and He ions will be used in the latter phases.

D. Sample Temperature

The effects of sample temperature during the irradiation process will be examined once the effects of the parameters discussed above are more clearly understood.